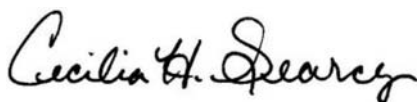EBERLINE
SERVICES

Eberline Services
Oak Ridge Laboratory
Analytical Procedure

AP-018

Operation of the Alpha Spectroscopy System

AUTHORIZATION AND APPROVAL STATEMENT

This **Eberline Services** - Oak Ridge Laboratory, Analytical Procedure,
"Operation of the Alpha Spectroscopy System"
is authorized and approved in its entirety by:

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Date: **October 31, 2010**

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1.0   SCOPE, PURPOSE, AND APPLICATION

1.1   This method establishes the procedures for general use and calibration of the Canberra AMS, VAX system used to obtain and analyze alpha spectra for samples containing single or multiple alpha-emitting radionuclides. The method also describes how specific radionuclide analyses are determined from the alpha spectral data.

1.2   The scope of this method is limited to the operation of the alpha spectroscopy counting instrumentation.

1.3   This procedure is applicable to all sample matrices that can be successfully prepared for alpha spectroscopy analysis.

2.0   DETECTION LIMITS

2.1   The samples for this procedure are counted using an alpha spectroscopy system. The anticipated detection limits for this procedure are as follows:

Isotope	Soil	*Water*
Am-241	0.2884 pCi/g	0.4774 pCi/l
Cm-244	0.3350 pCi/g	0.5156 pCi/l
Cm-247	0.3093 pCi/g	0.7520 pCi/l
Cm-248	0.1600 pCi/g	0.3558 pCi/l
Np-237	0.3136 pCi/g	0.4935 pCi/l
Pu-238	0.2082 pCi/g	0.3847 pCi/l
Pu-239	0.1709 pCi/g	0.3159 pCi/l
Th-228	0.1552 pCi/g	0.3639 pCi/l
Th-230	0.1756 pCi/g	0.4177 pCi/l
Th-232	0.1450 pCi/g	0.3388 pCi/l
U-234	0.2015 pCi/g	0.4223 pCi/l
U-235	0.1566 pCi/g	0.3283 pCi/l
U-236	0.1293 pCi/g	0.2774 pCi/l
U-238	0.1724 pCi/g	0.3664 pCi/l
Ra-226	0.28 pCi/g	0.78 pCi/l

2.2   The values listed above are based on a sample size of approximately one gram or one-half liter and a 180-minute count time.

2.3   These detection limits are historic averages. Individual sample detection limits may vary.

3.0   SUMMARY OF TEST METHOD

3.1   Alpha particles with discrete energies are emitted during the decay of some radionuclides. The number of alpha particles emitted per unit time is directly related to the disintegration rates of the radionuclides, and the energies of the alpha particles are characteristic of the radionuclides in a sample. By obtaining an alpha spectrum one can identify the radionuclides in a mixture and establish the relative amounts of each.

3.2   Alpha spectrometry is normally performed with silicon surface barrier or ion implant detectors (Those at the Eberline Services – Oak Ridge Laboratory are primarily ion implant detectors with a 25 mm diameter active area). This type of detector produces a current pulse whenever an alpha particle dissipates its energy in the sensitive volume of the detector. The amplitude of the current pulse is directly proportional to the energy of the alpha particle, and the number of alpha particles entering the sensitive volume of the detector per unit time is directly related to the disintegration rate of the sample. Pulses are amplified to give a voltage output that is proportional to the incident alpha particle energy. These voltage pulses are processed and stored by a multichannel analyzer for subsequent display and analysis.

3.3   Energy and efficiency calibrations will be performed after initial installation, annually, and following system maintenance that could affect the calibration. Energy and efficiency checks will be performed weekly, or whenever any problem is suspected. A National Institute of Standards and Technology (NIST) traceable standard approximating the sample geometry is counted in a reproducible shelf position, a spectrum collected, and subsequent analysis performed. The energy calibration/check is generated from the newly acquired data by plotting peak centroids versus actual energies of the standard. The efficiency calibration/check is attained by comparing the observed count rate of the calibration standard to the known decay corrected emission rate of the standard. This efficiency value is then used for the energy range of 3.5-6.5 MeV.

3.4   The checks for efficiency and energy, and a background determination will be performed at least weekly. Energy and efficiency calibrations are covered in section 12.1. The daily pulser check is covered in section 12.2. Background measurements are covered in section 12.3.

3.5   Each sample and QC sample spectrum shall be assessed for correctly chosen ROIs, acceptable spectral resolution, acceptable energy calibration and interferences with analyte and tracer ROIs.

4.0   DEFINITIONS

4.1   *Background* - those counts that can be observed and thereby allowed for by measuring a source that is identical to the unknown source in all respects except for the absence of radioactivity. These counts can be attributed to environmental radioactivity, recoil contamination of the detector, sample containers, cosmic rays, electronic noise pulses, etc.

4.2   Traceable Calibration Standard - a calibrated radioactive source, with stated accuracy, whose calibration is certified by or to NIST or an equivalent organization.

4.3   Efficiency - a percent of decay events from a standard radioactive source that are seen and measured by a detector.

4.4   *FWHM (Full Width Half Maximum)* - the full width of an alpha peak distribution measured at half the maximum peak height at the peak centroid

5.0   INTERFERENCES

None

6.0   SAFETY

Laboratory chemical and general safety shall be conducted as required within *"Eberline Services – Oak Ridge Laboratory, Site Specific Chemical Hygiene Plan", Latest Version*

Laboratory radiation safety shall be conducted as required within *"Eberline Services – Radiation Protection Plan and Attachments", Latest Version*

Waste management and sample return shall be conducted as required within *"Eberline Services – Waste Management Plan", Latest Version*

6.1   Housekeeping

6.1.1   All work areas shall be kept as clean as possible at all times and the entire work area shall be cleaned at the conclusion of the last shift of the day.

6.1.2   Minimize unnecessary items and clutter.

6.1.3   Promptly clean any spills that occur using the guidance contained in the Emergency Action Plan, Spill Response Procedure and support of the Radiation Safety Officer and Health and Safety Officer if necessary.

6.2   Clearly, label all sample containers (beakers, bottles, c-tubes etc.) with the work order number, analysis fraction, and analyte identification information such as "Total Sr", "Iso-U", or some other recognizable wording.

6.3   Any labels that identify the hazards associated with a particular sample container at the time of receipt will remain affixed to that container AND to ALL subsequent sub sampling from, and disposal of, that container.

6.4   Dispose of all waste in the appropriate containers as directed by the Waste Management Plan.

6.5   Dispose non-rad waste in appropriate containers, DO NOT PUT NON-RAD WASTE INTO RAD WASTE CONTAINERS.

6.6   Personal protective equipment for this procedure shall consist of a lab coat or protective apron, safety glasses or goggles and chemical resistant laboratory gloves.

7.0   EQUIPMENT AND SUPPLIES

7.1   Alpha spectrometer

7.2   PIPS (Passivated, Implanted Planar Silicone Detectors) or equivalent

7.3   Multichannel analyzer (VAX workstation or equivalent)

7.4   Vacuum pump

7.5   Remote parallel interface (RPI)

7.6   AMX analog multiplexer module

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7.7   Acquisition interface module

7.8   ADC (analog to digital converter)

7.9   The laboratory may use pre-cleaned disposable plastic lab ware as appropriate and applicable to this or any other analytical procedure. Disposable plastic ware will be disposed of in the appropriate waste container after use.

8.0   REAGENTS AND STANDARDS

8.1   NIST traceable calibration standard

8.2   Radioactive check sources

9.0   SAMPLE COLLECTION, PRESERVATION, AND STORAGE

9.1   Sample collection and preservation is not the responsibility of the laboratory and is not applicable to this procedure. Upon receipt of water samples, the laboratory may preserve/pH adjust the samples depending on the composition of the sample and the requested analysis.

9.2   Unless otherwise directed by the client, after receipt, all soil, solid, water, and vegetation samples will be segregated according to preliminary activity scans and stored in a secure, climate controlled location. Tissue sample will be stored in a freezer prior to analysis.

10.0   QUALITY CONTROL

10.1   One "Laboratory Control Sample (LCS)" shall be analyzed with every 20 samples. The LCS will be prepared and analyzed the same way and along with the analysis batch for the same analytical parameter.

10.2   One analysis blank shall be run with every 20 samples. If there are less than 20 samples per analysis batch, then one blank per batch shall be analyzed.

10.3   A minimum of one or a designated number of client samples shall be duplicated with every 20 samples (one sample for every 10 client samples will be duplicated for RCRA or SW846 analyses). If there are less than 20 samples per analysis batch, then a minimum of one or a sufficient number of duplicates to meet client criteria shall be analyzed per analytical batch. Where the matrix type, limited sample volume or other special considerations preclude this as a viable option, a replicate analysis will be used for QC evaluation.

10.4   If requested by a client, a matrix spike composed of a sample spiked with a standard containing at least one of the isotopes in question (NIST traceable or equivalent) shall be run with each batch.

10.5   Radioactive isotopic tracers are used for isotopic specific analysis by alpha spectrometry. The tracer is the same or similar element as the analyte so that it undergoes similar reactions during processing. Contribution to the analyte ROI(s) are evaluated, the method of correction discussed with the client, and corrected and documented if necessary.

10.6   Pulser checks shall be performed daily prior to counting samples.

10.7   Efficiency and energy calibrations shall be performed annually at a minimum, after instrument maintenance that may affect the calibration, or more frequently as needed. Efficiency determinations shall be performed when the check source count is outside the acceptable limits of the control chart.

10.8 Detector backgrounds, and efficiency and energy checks shall be performed once each calendar week. The gross counts in each target analyte and tracer ROI shall be corrected for the particular detector's background contribution in those same ROIs.

11.0 CALIBRATION AND STANDARDIZATION

11.1 The instrument calibration procedures are covered in the body of this procedure.

11.2 Eberline Services will use well characterized, NIST traceable alpha sources that will give a minimum of four alpha peaks that are well distributed over the range of approximately 3.5 to 6.5 MeV. It is desirable that there be no more than 10% difference in the levels of activity between the various alpha peaks. These sources should be electroplated on 25-mm steel discs and have the appropriate accompanying documentation showing, at a minimum, traceability to an NIST or equivalent standard along with the energies of the alpha particles emitted from the standard and the relative activity for each isotope.

11.3 Annual calibration standards are counted to collect at least net 3,000 counts under each ROI.

11.4 Weekly efficiency, energy and resolution calibration checks are counted to collect at least 2,000 net counts (approximately 3 hour count) under each ROI.

11.5 The dilution of NIST traceable (or equivalent) standard solutions is covered in procedure MP-009.

12.0 PROCEDURE

12.1 Efficiency, Energy and Resolution calibrations/checks.

Annual calibrations and weekly checks of energy, efficiency and resolution are processed in the same manner.

12.1.1 Load a standard into each counter. Close the chamber doors and start evacuation of the chambers. After the chambers have reached the operating pressure, turn on the detector bias supply for the chambers that still requires this action.

12.1.2 From the Main Menu select 1) "Count", which accesses the Alpha Counting drop-down menu.

12.1.3 From the Alpha Counting Menu select 4) "Primes" for a calibration, or 5) "Seconds" for a calibration check. The prompt. "Enter Banks e.g., "1,2,4-6,9" or "ALL" will appear.

NOTE
The software will frequently use the term "BANKS" when what it is actually referring to is detectors that are to be selected or have already been selected for use.

12.1.4 At the prompt, select appropriate detectors (i.e. 1-16, etc) then select "OK" by clicking on it with the mouse pointer.

12.1.5 The software should respond with the question "Is everything ready to count on banks:" and then list the banks containing the detectors selected for use. If all chambers are ready to use, select "OK" by clicking on it with the mouse pointer.

12.1.6 The sequence file is set up and counting should begin. Make the appropriate entries in the instrument log book to record the counting of the efficiency and energy calibration standards.

12.1.7 The Process begins and the results are shown on the video screen for each detector:

Energy Calibration:	Old	New
Offset:	(i.e., 3604.7)	(i.e., 3584)
Slope:	(i.e., 2-623)	(i.e. 2-657)
Quadratic:	(i.e., 2.47E-4)	(i.e., 2.31E-4)
ChiSquare:	(i.e., 0.00E+00)	(i.e., 5.11E+01)

In this case the slope should be less than 15 keV per channel for each detector. The equation with the slope and Y-intercept for the fit will be included in the annual calibration package.

12.1.8 Should the results be saved (YES/NO)? Choose the appropriate result (i.e., YES).

The FWHM and Efficiency results are displayed on the video monitor for each detector.

FWHM Calibration:	Old	New
Average (KeV)	(i.e., 17.51)	(i.e., 16.46)
Average (Chan)	(i.e., 6.52)	(i.e., 6.19)

The FWHM should be less than or equal to 70 keV for each detector.

Calculated Efficiencies:

Energy	Efficiency (%)
4824.20	26.904± 2.287
5155.40	25.109± 2.118
5480.35	24.277± 2.073
5796.11	23.855± 2.125

The peak centroid should be within ± 40 keV of the referenced energy value.

Efficiency Calibration	Old	New
Average (percent)	23.871	24.961
Uncertainty of Average	1.031%	1.073%

12.1.9 Should the results be saved (YES/NO)? Select the appropriate result (i.e., YES).

12.1.10 These results are displayed on the video monitor for each detector and the appropriate answers should be chosen until all the results for all detectors have been reviewed.

12.1.11 The QC reports and plots will be generated automatically. Review the printout for any out of control conditions. If any of the data is out of control, contact the Count Room Supervisor immediately. The control limits are based upon 2 and 3 standard deviations of historical data.

12.2 Daily pulser checks

This pulser check is performed daily prior to counting samples to verify the proper operation of the detectors. Peak centroid, Peak Energy Pulser count rate, and peak FWHM are monitored and stored in quality assurance files.

12.2.1 Ensure that all detectors are empty. Close the chamber doors and start evacuation of the chambers.

12.2.2 After the chambers have reached the operating pressure, switch on the bias voltage on the detectors that still require this.

12.2.3 Select 1) "Count" to access the Alpha Counting Menu.

12.2.4 Select 3), "Pulsers".

NOTE
The software will frequently use the term "BANKS" when what it is actually referring to is detectors that are to be selected or have already been selected for use.

12.2.5 At the prompt, "Enter banks e.g. 1,2,4-6, 9, or ALL", enter the appropriate number.

12.2.6 The software should respond with the question "Is everything ready to count on banks:" and then list the banks containing the detectors selected for use. If all chambers are ready to use, select "OK" by clicking on it with the mouse pointer.

12.2.7 The sequence file is set up and counting should begin. Make the appropriate entries in the instrument log book to record the pulser check.

12.2.8 The sequence file is set up and counting should begin. Make the appropriate entries in the instrument log book to record the counting of the efficiency and energy calibration standards.

12.2.9 This accesses the following choices:

Detector List	leave blank
Items to process	enter "Da" for daily pulser
Sample batch name	leave blank
Sample Actinide	Leave blank

12.2.10 The files are then automatically purged and the program will proceed to automatically process the pulser data one detector at a time. After the data is processed (GRAPHS FOR FWHM, PEAK ENERGY, CTS/SEC., AND PEAK CENTROID), a QC report in tabular form will be generated documenting the results of the check.

12.2.11 Review the printout for any out of control conditions. If any of the data is out of control, contact the Count Room Supervisor immediately. The criteria for control limits are based upon two and three-sigma control levels for the particular parameters listed above.

12.3 Weekly Background

The gross counts in each target analyte and tracer ROI is corrected for the particular detector's background contribution in those same ROIs.

12.3.1 Ensure that all detectors are empty. Close the chamber doors and start evacuation of the chambers.

12.3.2 After the chambers have reached the operating pressure, switch on the bias voltage on the cells that still require this.

12.3.3 From the Main Menu select 1) Count, which accesses the Alpha Counting Menu.

12.3.4 From the Alpha Counting Menu select 1) Background, which access the prompt, "Enter banks e.g. 1,2,4-6, 9, or ALL", enter the appropriate response.

12.3.5 The software should respond with the question "Is everything ready to count on banks:" and then list the banks containing the detectors selected for use. If all chambers are ready to use, select "OK" by clicking on it with the mouse pointer.

12.3.6 The sequence file is set up and counting should begin. Make the appropriate entries in the instrument log-book to record the counting of the background.

12.3.7 The system will count the backgrounds for 1000 minutes, which is significantly longer than sample count times.

12.3.8 After the data has been processed, a report will be printed in tabular form, documenting the results. Review the printout for any out-of-control conditions. If any of the data is out of control, contact the Count Room Supervisor immediately.

12.3.9 Background control limits will be set based on two and three standard deviations from the mean for the entire energy range (approximately 3.5 to 6.5 MeV), and shall not exceed 100 counts over the entire spectrum. It should be noted that a spurious low background count rate, by itself, will not be considered a basis for putting the detector out of service.

NOTE

The technician processing the background counts shall review each spectrum for anomalous peaks. This review is essential to ensure that background counts are not accumulating under an individual ROI. If any peaks appear anomalous, notify the count room supervisor.

12.4 Sample counting

12.4.1 Ensure the bias supply to the detector is off and vent the alpha spectrometer sample chamber to atmosphere by placing the "pump/vent" switch on the spectrometer to the "vent position".

12.4.2 Open the door of the sample chamber. Carefully remove any sample that is in the chamber with a pair of tweezers and place it in a proper storage container.

12.4.3 Using a pair of tweezers, carefully position the next sample that is to be counted in the sample holder then center the sample beneath the detector face and ensure the sample shelf is in the proper location.

12.4.4 Close the door on each sample chamber and position the vacuum manifold selector switch that corresponds to that sample chamber, to the pump position and begin to evacuate the chamber. Continue to pump the chamber until a pressure of 500 microns or less is reached as indicated by the vacuum gauge.

12.4.5 For bank one turn on the detector bias voltage supply and verify the proper operating bias by selecting the "bias" position with function selector switch on the front of the alpha spectrometer, or the "on" position for bank two (bank three is controlled through the software.

12.4.6 Select "Count", to access the alpha counting menu.

12.4.7 Select "Samples".

NOTE

The software will frequently use the term "BANKS" when what it is actually referring to is detectors that are to be selected or have already been selected for use.

12.4.8 At the prompt, "Enter banks (meaning detectors) e.g. 1,2,4-6, 9, or ALL", select the detectors being used and click on "OK" with the mouse pointer.

12.4.9 The Default Sample Setup Menu will be displayed. This information is applicable to the entire analytical batch. Enter the applicable information in the appropriate boxes:

- Batch Number
- Alt. Batch Number
- Analysis Library Code
- Sample Matrix
- Collection Date/Time
- Preset Count Time
- Sample Prep % Error
- N Sigma Multiplier
- Activity Units
- Units
- Tracer Lambda
- Tracer Cer. ID
- MDA LLD Constant
- MDA Confidence Level

- (Internal Work Order Number, run, and analysis code)
- (Leave Blank)
- (UU for Uranium etc.)
- (Water, Soil, etc.)
- (Most common one for the batch)
- (Normally 2 hours & 50 minutes)
- (Normally "3.0")
- (Normally "2.0")
- ("pCi" or other from menu)
- ("grams", "liters" etc. from menu)
- (leave blank here)
- (Tracer ID Number)
- ("2.710")
- ("4.65")

After entering the above values, review the values and if all are correct select "OK" at the bottom of the page.

12.4.10 After the batch information is entered, a box will appear allowing you to enter the information for the first sample. Fill in the boxes as indicated below.

- Sample Quantity   (Sample aliquot)

NOTE

In some cases where the sample aliquot is very small (e-10 or smaller), leave this at the default value and when all other data on the screen has been entered and "OK" has been selected, go to the Spectrum Screen and select "Edit" at the top of the screen and you can then enter the aliquot in the dialog box that appears.

- Detector Number
- Chamber Contents Code
- Sample ID
- Batch Number
- Alt Batch Number
- Sample Title
- Sample Date/Time
- Sample Prep % Error
- N-Sigma Error Multiplier
- Activity Units
- Units
- Tracer Lambda
- Tracer ID

- (should be filled in already)
- (Dropdown menu to select sample type)
- (Must be the Two-digit analysis fraction number)
- (This should be already filled in)
- (leave this blank)
- (Type in as much of Client ID# as possible)
- (Collection Date/Time for sample)
- ("3.00" if not already there)
- ("2.0" if not already there)
- (Should be OK from the batch entries)
- (Should be OK from the batch entries)
- (Tracer volume in microliters/milligrams)
- (Should already be there)

- Tracerless recovery
- (Only enter a value for Ra-226 or Np-237 analyses)

12.4.11 After the first sample is entered, the system will ask "Is EVERYTHING ready to count on BANKS:" (lists detectors already selected). Select "OK" by clicking on the box with the mouse pointer.

12.4.12 The system will now bring up an identical set of entries for the next sample. Repeat the entries changing the sample time, tracer volume, etc as necessary.

12.4.13 After entering the data for the last sample the system will stop asking for additional sample information and will automatically start counting the samples.

12.4.14 Enter the appropriate information into the log book to record the counting of the sample work order.

12.5 Processing sample data

Visually review the data collected. Each spectrum should contain an individual sample spectrum corresponding to the sample loaded in that particular chamber. If any of the spectra are missing or appear to be wrong, contact the Count Room Supervisor.

12.5.1 If the sample shows more than 5000 counts in any single region, contact the count room supervisor to see if it will be necessary to perform a background check on the diode prior to counting additional client samples on this particular detector.

12.5.2 From the Main Menu select "Process" (a pull-down menu) and under that select "Create Names.dat" which generates another pull-down menu and from that menu select "Samples."

12.5.3 The system will create an information box on the screen. In the area designated "For Samples Only" enter the batch name that was created in 12.5.1.9 and also enter the analysis code in the "Actinide" entry. Select "OK" to end this screen.

12.5.4 The screen will now show a list of files. If the batch number and number of samples in the batch agree with the information in the folder, this is the data to process. Click on "OK" to continue processing. If they don't match, there is likely an error in the batch name. Select "Cancel" and return to step 12.5.1 and start over again.

12.5.5 The first sample spectrum will automatically be displayed in the bottom of the next window. After each spectrum is displayed answer the question "Do the standard regions look ok? The options are "OK" or "Cancel". If the regions are acceptable – the peaks highlighted generally appear to be free from interference, do not exhibit excessive trailing or shifting etc., select "OK" and proceed to step 12.5.2.17. The Radium-226 spectra will be accepted regardless of appearance and will be evaluated later.

12.5.6 If there is a problem with the spectrum, select "Cancel". This action will bring up a dialog box that has the text "Use Manual Regions". Select "OK".

12.5.7 Switch off the Caps Lock on the keyboard.

12.5.8 On the spectrum screen, click on the ROI index ("-" or "+") until the markers enclose the region you want to change.

12.5.9 Press the "Remove" key on the keyboard.

12.5.10 Left click on the marker and drag it to where you what one side of the new region of interest. Repeat with the other maker.

12.5.11 Press the "Insert Here" key on the keyboard.

12.5.12 Repeat for any other regions that need to be adjusted.

12.5.13 The top of the processing window (not the spectrum screen) has the dialog box "Continue when new regions are created". When all of the regions are satisfactory, select "OK".

12.5.14 A new dialog box will appear asking "Do Manual Regions Look OK". Select "OK".

12.5.15 The system will bring up the next sample spectrum for examination.

12.5.16 After the sample processing is completed the system will return to the Alpha Counting Menu.

12.5.17 Data is saved and retained on the system. The count room supervisor periodically copies the data to tape and deletes the data from the system in order to free memory for processing. The tapes are designated by date range and stored in a fire-proof cabinet with the other LIMS backup data. Data, including spectra, can be regenerated from these tape backups.

13.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

13.1 All excess sample materials, extracts, byproducts, and associated waste will be disposed of in the appropriate containers and segregated into the appropriate waste streams for final disposal according to the Waste Management Plan, WMP-01.

13.2 All laboratory activities associated with this procedure will be carried out in the fashion designed to generate the least amount of waste possible and still achieve the necessary quality of data.

13.3 Pre-cleaned disposable plastic lab ware will be placed in the appropriate waste container following its use in the laboratory.

14.0 CALCULATIONS

14.1 Efficiency

$$Eff = \frac{Cpm_o - Cpm_b}{Dpm_c}$$

Where:
- Eff = Efficiency
- Cpm_o = observed counts per minute of the standard
- Cpm_b = background counts per minute
- Dpm_c = decay corrected disintegration rate of the certified calibration standard

14.2 Decay corrections

$$A_f = A_i\, e^{-\frac{\ln 2}{t_{1/2}}T}$$

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Where:

A_f = Decay corrected activity
A_i = Initial activity
e = Exponential function $\approx$ 2.71828
$t_{1/2}$ = Half-life of the radionuclide
In2   = Natural log of 2 = 0.693
T   = Time of decay

14.3   Counting Uncertainty

$$CU = \frac{1.96\sqrt{\dfrac{Cpm_s}{T_s} + \dfrac{Cpm_b}{T_b}}}{Eff * V * D * A * 2.22}$$

Where:

CU   = Counting Uncertainty (sometimes referred to as "error")
1.96   = Conversion to two-sigma (95.5%) Uncertainty
Cpm_s = Counts per minute sample
Cpm_b = Counts per minute background
T_s = Count time, minutes, sample
T_b = Count time, minutes, background
Eff   = Counting efficiency
V   = Sample volume/weight
D   = Radioactive Ingrowth or Decay Factors as Necessary
A   = Abundance
2.22   = Dpm to Pico Curies conversion factor

14.4   Chemical yield (Y)

$$Y = \frac{C_T}{Eff * T * Dpm_T}$$

Where:

C_T = Total *Net* Counts in the Tracer Peak (tracer – bkg).
Eff   = Detector Efficiency.
T   = Count Time (in minutes).
Dpm_T = Dpm of Tracer added to each sample.

14.5   Minimum Detectable Activity (MDA) calculation

$$MDA = \frac{2.71 + 4.65\sqrt{Cts_B}}{Eff * Y * A * V * T_S}$$

Where:

2.71   =   Statistical Factor (95% Confidence Level).
4.65   =   Confidence Factor (95% Confidence Level).
Cts_B =   Background Counts.
2.22   =   Dpm to picoCuries conversion factor.
Eff   =   Detector Efficiency.
V   =   Sample Volume/Weight.

Y   =   Chemical Yield.
T_S =   Sample Count Time (in minutes).
A   =   Decay Abundance.

14.6   Activity calculation

$$Activity(pCi/unit) = \frac{C_s - C_b}{Eff * Y * V * D * A * T_s * 2.22}$$

Where:

C_s = Total counts in peak region
C_b = Total background in peak region
Eff   = Counting efficiency
Y   = Yield
V   = Sample volume/weight
D   = Radioactive Ingrowth or Decay Factors as Necessary
A   = Abundance
T_s = Sample count time (in minutes)
2.22   = Dpm to Pico Curies conversion factor

14.7   Normalized Difference

For duplicate/replicate analyses where the results are relatively close to the detection limits, the normalized difference comparison is an appropriate method for determining the statistical agreement of the two values.

$$Normalized\,Difference = \frac{\left| Result_1 - Result_2 \right|}{\sqrt{Uncertainty_1^2 + Uncertainty_2^2}}$$

14.8   Relative Percent Difference

The relative percent difference calculation is generally useful when two duplicate/replicate results are significantly elevated above their detection limits.

$$Relative\,Percent\,Difference = \frac{\left| Result_1 - Result_2 \right|}{Result_1 - Result_2 / 2} * 100$$

14.9   Combined Standard Uncertainty

In order to incorporate indeterminate errors such as having more than one technician involved in the analysis of the samples or the same operation performed by different personnel on different days, Eberline Services uses the historic data for the analysis of known laboratory control standards to determine the combined standard total uncertainty involved in the analysis of a given isotope.

$$CSU = \sqrt{CU^2 + Activity^2 \left(TPU_F\right)^2}$$

Where:

CSU   = Combined Standard Uncertainty
CU   = Counting Uncertainty
Activity   = Activity of the sample

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$$\text{TPU}_f = \text{Total Propagated Uncertainty factor} = \frac{\sigma_{LCS}}{\text{Known LCS Value}}$$

LCS = Laboratory Control Sample

NOTE: The CSU was derived from the following TPU equation

$$\text{TPU} = \sqrt{\left(\frac{(\text{St.Dev. X 1.96})}{\text{Avg.Known}}\right)^2 + \left(\frac{\text{Count.Unct.}}{\text{Activity}}\right)^2} \text{ X Activity}$$

15.0   METHOD PERFORMANCE

15.1   The initial method performance shall be determined using the method detailed in procedure MP-028.

15.2   The continuing method performance is monitored through the use to the laboratory control standards, blanks and duplicates/replicates.

16.0   DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASUREMENTS

It is the laboratory policy to analyze a Laboratory Control Sample (LCS), a Laboratory Method Blank (MBL), and a Duplicate (DUP) with each work order. Soil samples will be reported on a dry weight basis unless otherwise requested by the client. Work orders are unique for each client, matrix, and isotope. Specific client requirements may supersede the following laboratory default criteria.

16.1   The default, laboratory limits for evaluating the performance of the Laboratory Control Standard is the 75% to 125% recovery range or a normalized difference of <3.0 for low activity standards (U-235). Generally, we have found that these limits are equal to or more restrictive than three standard deviations from the mean. Results within this range are acceptable; results that are outside this range either require reanalysis or explanation in the sample case narrative portion of the data report.

16.2   Counting uncertainties are estimated by the square root of counts except when there are zero (0) counts. In the case of zero (0) counts, the value under the radical is assumed to be one count.

16.3   The blank result should be less than or equal to 2 times the Combined Standard Uncertainty (CSU). If the blank result is greater than 2 times the CSU, the samples in the work order may require reanalysis or the situation should be explained in the case narrative section of the data report. If blanks containing Naturally Occurring Radioactive Material (NORM) are >2 times the CSU, they will be evaluated against typical background activity levels and noted as such in the case narrative.

16.4   The duplicate/replicate analyses are compared using either the normalized difference comparison or relative percent difference. The normalized difference result should be less than or equal to a value of 3.0 and the relative percent difference result should be less than or equal to 25%. When either the relative percent difference or the normalized difference pass, then the duplicate is acceptable. If the duplicate/replicate fails both tests, the samples in the work order may require reanalysis or the situation will be explained in the case narrative section of the data report. Relative percent difference and normalized difference for non-positive, background equivalent or noise equivalent results will be discussed in the case narrative, but not be grounds for re-analysis.

16.5   The default laboratory limits for acceptance or rejection of the Matrix Spike are 60% to 140%. Results within this range are acceptable, results that are outside this range are not acceptable and either requires reanalysis or explanation in the sample case narrative portion of the data report.

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16.6   The limits for tracer recovery of any sample are 30% to 110%. The limits for carrier recovery are 40% to 110%. Analytical results within this range will be calculated using the determined chemical recovery. Samples with tracer or carrier recoveries outside this range will be reanalyzed or the situation will be thoroughly explained in the client case narrative.

16.7   RCRA Method 9315 (Alpha Emitting Radium Isotopes) requireS a sample duplicate be analyzed at a frequency of one in every ten samples.

16.8   Additional criteria specific to alpha spectrometry include:

16.8.1   If the FWHM for the tracer peak exceeds 100 keV and/or the peak energy does not fall within $\pm$ 50keV of the known peak energy.

16.8.2   Target analyte or tracer peaks are not resolved (due to activity of sample).

16.8.3   Significant interferences that impact the analyte and/or target ROI(s).

16.9   Daily Pulser Checks

16.9.1   Pulser check will be performed each day of operation. Each detector result will be examined for the presence or absence of a pulser peak. If a pulser peak is not present, the detector will be placed out of service.

16.9.2   The pulser FWHM, and centroid location are monitored based on fixed limits. The FWHM should be less than 75 keV and the centroid should be within 50 keV of the anticipated location.

16.9.3   If the result is outside of the control limit, it may be rechecked and if acceptable, the detector may be used that day. If the detector fails a second check, it will be placed out of service, noted as so in the logbook and the Count Room Supervisor will be notified in a timely manner.

16.10   Weekly Background

Background determination/checks will be performed each week. The background results will be compared to a fixed limit of 100 counts in 1000 minutes. The technician processing the background counts shall review each spectrum for anomalous peaks. This review is essential to ensure that background counts are not accumulating under an individual ROI. If any peaks appear anomalous, notify the count room supervisor.

16.10.1 If the background is within the administrative control limits. The detector is acceptable for use and no action is required.

16.10.2 If the background is outside of the upper three-sigma limit (or greater than 100 counts in the 1000 minute count time), the detector will be placed out of service until the background can be checked again. If a second background is performed immediately after the first and this count also fails, the detector will be placed out of service and the Count Room Supervisor should be contacted to determine the necessary action.

16.10.3 It should be noted that a background that falls outside of the lower three-sigma limit is not necessarily a basis for putting the detector out of service.

16.11   Efficiency, Energy, and Full-Width Half-Maximum (FWHM) Checks

These parameters will be checked on a weekly basis. The efficiency for each diode will be checked each week. The efficiency of the electroplated standards will be plotted against two and three standard deviations of the historical mean since the time of calibration.

6.11.1   If the efficiency is within two standard deviations of the mean, no action is necessary.

6.11.2   If the efficiency is between two and three standard deviations of the mean, the "investigate" region, the detector may be used but at a minimum the situation must be noted in the instrument logbook.

6.11.3   If the efficiency is outside of the three-sigma limit, the detector may be immediately rechecked. If the second check fails, the detector should be placed out of service and the Count Room Supervisor notified to determine what further action might be necessary..

6.11.4   The weekly efficiency is used to monitor instrument performance, and is not used to calculate results.

6.11.5   The FWHM and centroid location are monitored based on fixed limits. The FWHM should be less than 70 keV and the centroid should be within 40 keV of the anticipated location.

16.12   Plotting of Results Outside the Three-Sigma Limit

16.12.1 In some cases, it may be necessary to remove data points that adversely affect the QA/QC charts. While there should be an occasional point outside the three-sigma limit due to simple statistics (approximately three for every 1000 measurements) each point that falls outside the three-sigma should be investigated to determine if it should be included in the population used to generate the control limits.

16.12.2 All QA/QC data point will be retained in the database regardless of whether or not they are in control.

17.0   CORRECTIVE ACTIONS FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Sample data which is deemed to be unacceptable will be reanalyzed according to the criteria in MP-030, Quality Assurance Implementation Plan, Section 16.0, when it is not possible to relate the deficiency to a calculational or clerical error and there is sufficient sample available for reanalysis.

18.0   REFERENCES

18.1   "1990 Annual Book of ASTM Standards", Volume 12.02, E181.

18.2   Canberra NDSP Sample Counting Software.

18.3   Krieger, Herman L. and Whittaker, Earl L.; Prescribed Procedures for the Measurement of Radioactivity in Drinking Water. USEPA, EPA-600/4-80-032, August 1980.

19.0   TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

19.1   Validation data is available on file.

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19.2   There are no tables or diagrams associated with this procedure.

20.0   DEVIATIONS FROM REFERENCED METHODS

Because this procedure as written applies to a variety of isotopes prepared by numerous procedures, the references presented above may not have a specific step-by-step correlation for any specific analytical method contained in one of the above reference to the procedure as written for the operation of the instrumentation.